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13. ABSTRACT (Maximum 200 words) Fabrication of a metal-based intelligent composite with shape memory alloy (SMA) wires was investigated. Composition gradient films of Al <sub>2</sub> O <sub>3</sub> or Ta <sub>x</sub> O <sub>y</sub> N <sub>z</sub> can be coated on the NiTi wire by the ion-implantation, ion-sputtering deposition, and high-temperature oxidation technique. Test results indicate that such films could have very good adhesion with the NiTi wire and allow the SMA wires and metal matrix to have good insulation properties. The vibration test further demonstrates that the aluminum-based composite-embedded with the NiTi SMA wire could significantly reduce vibration.				
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# **FOURTH INTERNATIONAL CONFERENCE ON ADAPTIVE STRUCTURES**

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# A METAL BASED INTELLIGENT COMPOSITE WITH SMA MATERIALS

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## ABSTRACT

An investigation has been conducted on the fabrication of a metal based intelligent composite with shape memory alloy (SMA) wires. The results show that composition gradient films of  $\text{Al}_2\text{O}_3$  or  $\text{Ta}_x\text{O}_y\text{N}_z$  can be coated on the NiTi wire by the ion-implantation, ion-sputtering deposition, and high-temperature oxidation technique. Test results indicate that such films could have very good adhesion with the NiTi wire and allow the SMA wires and metal matrix to have good insulation properties. The vibration test further demonstrates that the aluminum-based composite embedded with the NiTi SMA wire could significantly reduce vibration.

## INTRODUCTION

The NiTi shape memory alloy (SMA) has been recognized as an important intelligent/smart material and has been widely used in adaptive structures that utilize heat-activated, reversible crystallized phase transformation to generate high recovery stress, high stiffness, and a significant variation of electric resistance [1]. Some theoretical modeling [2-3] and experimental results [4-5] have shown that the NiTi SMA can be used to embed in a variety of structural materials to form adaptive structures for vibration and shape control. In the past, most research in this area has been limited to theoretical modeling and simulation. Only a few studies have been carried out on the experimental investigation of SMA embedded composites or structures [4-5], and then, only on the polymer-based composites or structures. It is anticipated that, in some circumstances, the metal matrix-based smart structure will be required for actual applications. This leads to the necessity of developing SMA-embedded metal-matrix composites. Thus far, there is limited literature dealing with this kind of material. Comparing the SMA-embedded composite based on a polymer matrix, the fabrication of a metal matrix-based composite is much more difficult. The great difficulty lies in the fabrication of an interface layer that can provide good adhesion, as well as good

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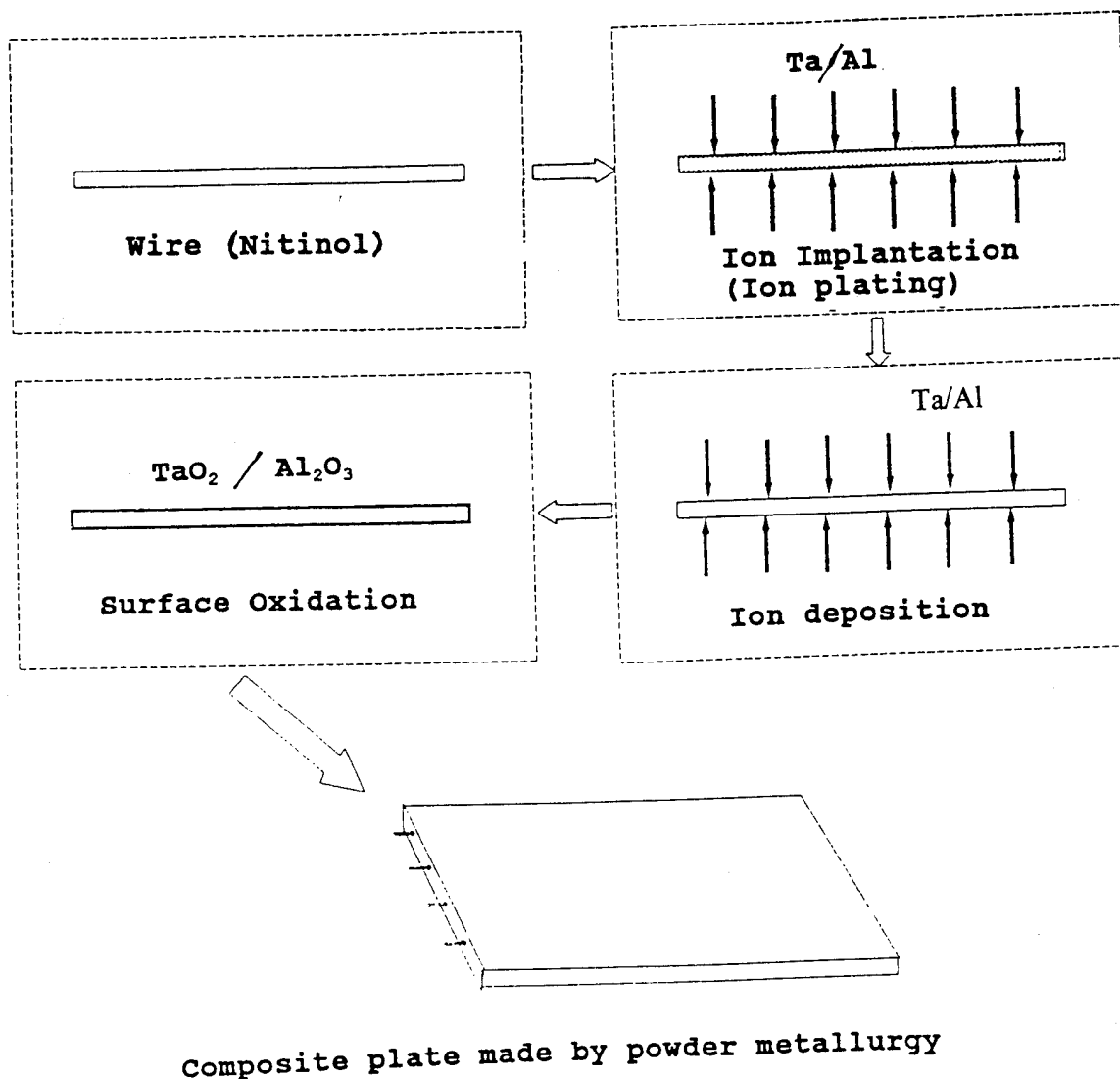


Figure 1. Schematic representation of the processing of the SMA-based composite plate.

insulation between the SMA wire and the metal matrix, since an electric current is needed to activate the phase transformation within the SMA wire. Since the phase transformation of the SMA involves a great deal of volume change, the mismatch in strain between the wire and the matrix is expected to be large. Therefore, providing a good insulation interface layer and ensuring that the layer can withstand hundreds and thousands of strain cycles is a big challenge. This may be why, up to the present, no research work on this type of material has been reported. This study intends to look into this challenging area, and the results of our primary investigation on the fabrication and characterization of the SMA-embedded metal-based composite, and the vibration suppression behavior of this kind of material will be reported and discussed.

## EXPERIMENTAL PROCEDURES

Fabrication of the SMA-embedded metal matrix-based composite is divided into two steps: (1) modification of the surface of the NiTi wire to form an insulation thin film; (2) fabrication of the composite by hot-press sintering the aluminum with the embedded NiTi wire. Aluminum is chosen in this study as the matrix material due to its low melting temperature and wide application in industry. Two kinds of insulation materials,  $\text{Al}_2\text{O}_3$  and Ta-containing oxide, were selected as an insulating thin film. The NiTi wire coated with  $\text{Al}_2\text{O}_3$  film was fabricated by the following process: (1) aluminum is implanted into the surface of the NiTi wire by the ion-implantation method with an implantation dose of  $2 \times 10^{17}$  ion/cm<sup>2</sup> to form a composition gradient transmission zone; (2) an aluminum film with a thickness of 1–10 nm was then deposited on the surface using the vapor plating method; (3) finally, a  $\text{Al}_2\text{O}_3$  film was formed by the electric-chemical anode oxidation of the Al film produced in step (2).

The NiTi wire coated with Ta-containing oxide film was fabricated by the following steps: (1) a TaN film was first formed by the tri-anode sputtering ion plating method under an  $\text{N}_2$  environment because of the implantation effect of the ion-beam. This process also provides a composition gradient transmission zone; (2) the TaN film was then oxidated at a high temperature to form a Ta-containing oxide film.

The complete composite was finally fabricated by a hot-press sintering method. First, the NiTi wire coated with  $\text{Al}_2\text{O}_3$  or Ta-containing oxide film was placed in the aluminum powder, and was then cold-pressed to 60% density. Finally, the aluminum composite with the NiTi wire was hot-pressed at 853K at a 150 MPa.

Figure 2 summarizes the entire process of the fabrication. The adhesion behavior of the NiTi wire and insulator film was characterized by the scratch method [6]. Distribution of the composition between the insulator film and the NiTi wire was measured by auger electron microscopy. The microstructure and composition of the SMA-embedded composite matrix was analyzed by scanning electron microscopy (SEM).

The vibration characteristics of the SMA-embedded aluminum composite was then tested by a fixed-end beam under a forced vibration condition. Figure 2 illustrates the setup of the vibration test.

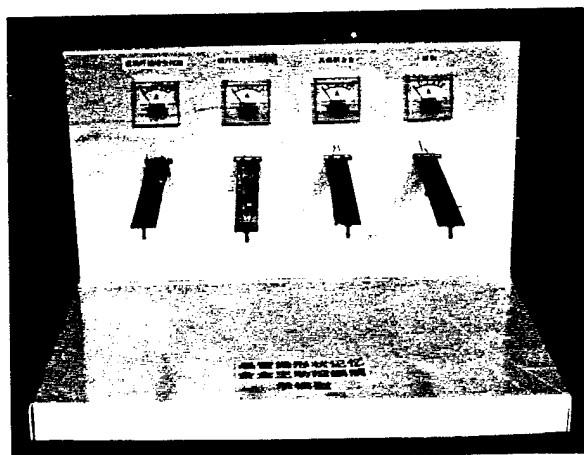


Figure 2. A photograph of the vibration test equipment.

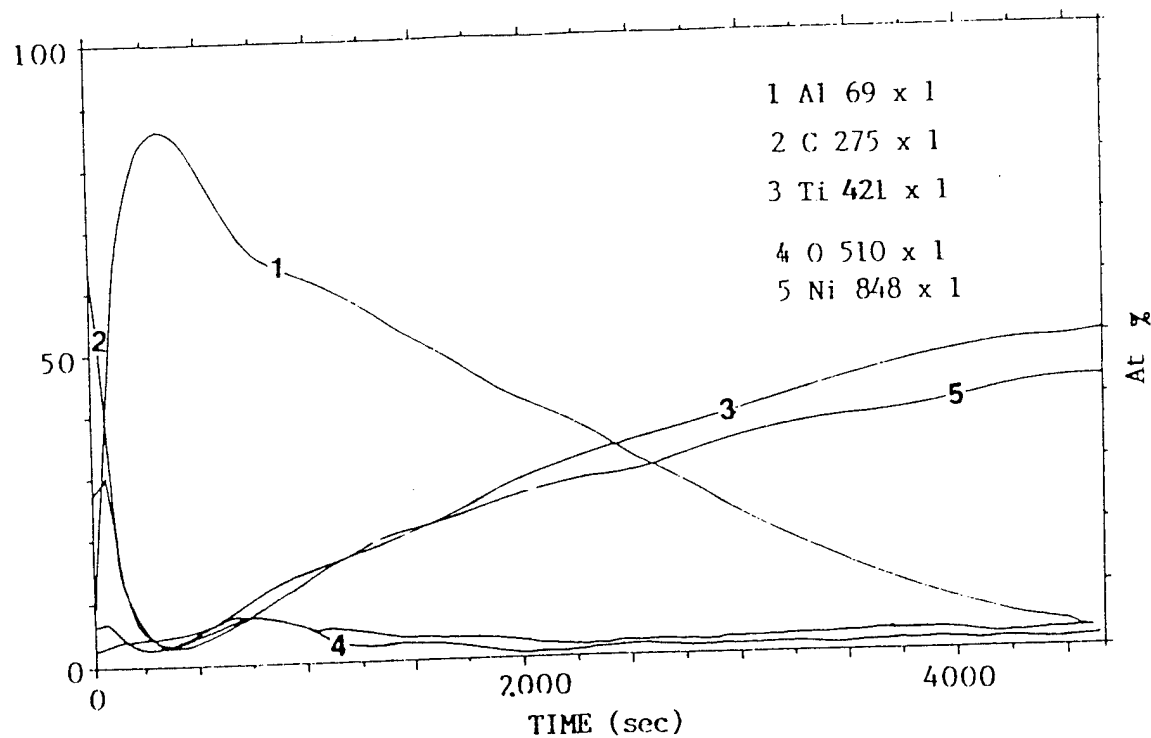


Figure 3. The composition distribution from the  $\text{Al}_2\text{O}_3$  film to NiTi wire analyzed by an Auger electron microscope.

## EXPERIMENTAL RESULT

### THE $\text{Al}_2\text{O}_3$ INSULATOR FILM

In order to obtain good adhesion between the insulation film and the NiTi wire, a composition gradient transmission zone was first formed by the ion-implantation method. Figure 3 shows the composition distribution analyzed by auger electron microscopy. It is readily seen that there is a transmission zone from the surface of the film to the NiTi wire. The scratch test conventionally used to determine the bonding strength of a thin film to a substrate was performed to check the adhesion strength of the  $\text{Al}_2\text{O}_3$  film and NiTi wire. The results are shown in Fig. 4. Based on the test, the critical load,  $P_c$ , required to scribe away the film is characterized by a sudden drop of  $F_y$ , see Fig. 4, and, for the  $\text{Al}_2\text{O}_3$  film, this turns out to be 1700 g. This relatively high value of  $P_c$  indicates a good adhesion between the  $\text{Al}_2\text{O}_3$  film and the NiTi wire. Meanwhile, the smooth curve in Fig. 4 also demonstrates that the film has good toughness. The good adhesion of the film and the wire may be attributed to the existence of a composition gradient zone.

An electric resistance measurement was also performed to determine the insulation properties of the interface between the NiTi wire and the  $\text{Al}_2\text{O}_3$  film. The value of the electric resistance was 0 ohm between the interior and the exterior of the wire before the film was deposited, and was more than 2000K $\Omega$  thereafter. This result demonstrates that the  $\text{Al}_2\text{O}_3$  film could successfully provide very good insulation between the wire and the matrix.

### THE TA-CONTAINING OXIDE FILM

The characteristics of the Ta-containing film is similar to that of the  $\text{Al}_2\text{O}_3$  film. Figure 5 depicts the composition gradient from the film surface to the NiTi wire. As can be seen in this figure, a composition gradient transmission zone clearly exists. The scratch technique was used again to test the adhesion behavior of the film to the NiTi wire. Figure 6 shows the test results. The critical load for debonding the film is calculated to be 1500g, which is on the same level as that of the  $\text{Al}_2\text{O}_3$  film (1700g). This indicates that the Ta-containing oxide film also has good adhesion with the NiTi wire. The smooth curve in Fig. 6 suggests that the Ta-containing film also has good toughness.

The resistance measurement was conducted to test the insulation characteristics of the Ta-containing film. The results show that the resistance of the interface between the NiTi and the film is zero before high temperature oxidation and 2000 K $\Omega$  after the process. These results indicate that (1) the oxide,  $\text{Ti}_x\text{O}_y\text{N}_z$ , can provide satisfactory insulation; and (2) the TiN is a conductor.

### THE ALUMINUM-BASED COMPOSITE WITH SMA

The SMA-embedded aluminum-based composite was synthesized by the powder metallurgy method. The detailed sintering process has been described in the

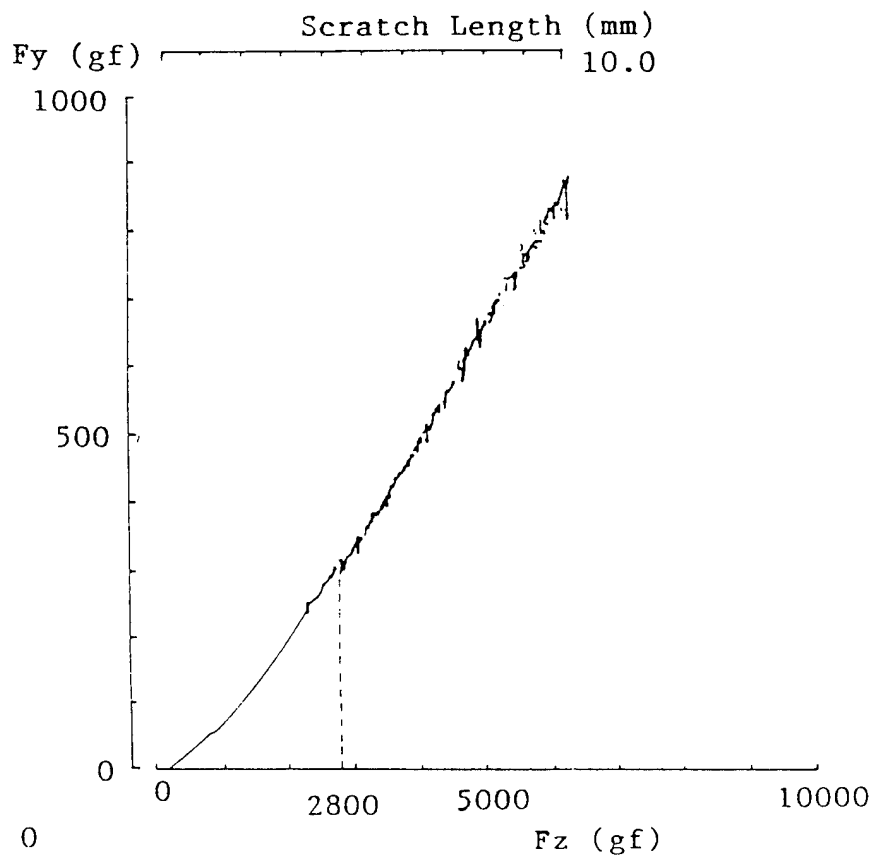


Figure 4. The scratch test on the wire coated  $\text{Al}_2\text{O}_3$  film.

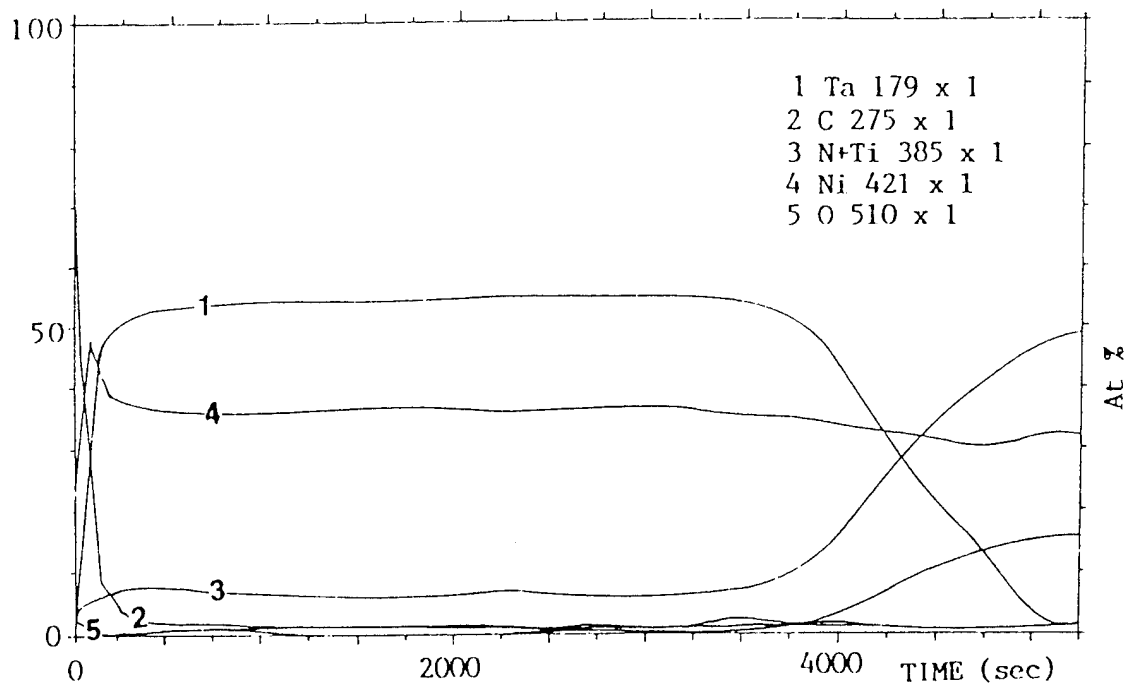


Figure 5. The composition distribution from the Ta-containing oxide film to NiTi wire analyzed by an Auger electron microscope.



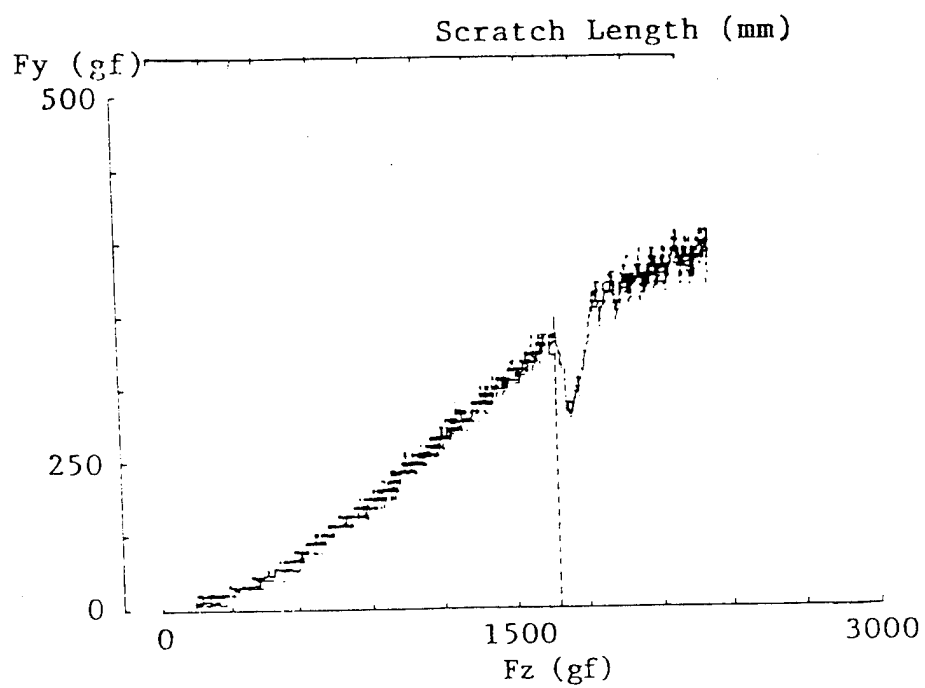


Figure 6. The scratch test on the NiTi wire coated Ta-containing oxide film.

experimental procedures. This section focuses on the microstructure characterization of this type of composite. Figure 7 shows the microstructure of the composites with the SMA,  $\text{Al}_2\text{O}_3$  interface, and the aluminum matrix. Figure 7a is a low magnification optical microstructure; Figure 7b shows the high magnification photographs at the boundary of the aluminum matrix, the  $\text{Al}_2\text{O}_3$  film, and the SMA. This photograph demonstrates that the  $\text{Al}_2\text{O}_3$  film can be maintained intact during the hot-press synthesis process. The oxygen content of the interface area, as determined by the X-ray energy dispersion spectrum (EDS), is illustrated in Fig. 7c. It clearly indicates, as expected, that the oxygen is maximum at the boundary.

The microstructure of the composite consists of the aluminum, the Ta-containing oxide film, and the SMA. Figure 8 shows the microstructure of the composite. It can be seen that this photograph is similar to Fig. 7. The results suggest that both  $\text{Al}_2\text{O}_3$  and Ta-containing foils can be successfully fabricated through various deposition methods.

### THE VIBRATION TEST

In order to determine the vibration behavior of the metal-based SMA composite, a simple cantilever beam under a forced vibration test was conducted. The arrangement of the experiment is demonstrated in Fig. 2. The samples used in the vibration test consist of a high-strength aluminum matrix and 0.5 mm diameter equiatomic NiTi wires. The  $A_s$  (austenite starting) transformation temperature of the NiTi wire is  $75^\circ\text{C}$ . Figure 9 shows the vibration spectrum after activation. Sample A is a high-strength aluminum alloy without any SMA wires; Sample B is the composite of the same matrix as Sample A, however, with SMA wires. The NiTi wires were heated to above the  $A_s$  transformation temperature. Figure 9 clearly shows a significant reduction in vibration as a consequence of the embedded SMA wire in Sample B. The phase transformation of the SMA varies the elastic modulus and absorbs energy during transformation. These help to damp out the vibration.

### RESULTS AND DISCUSSION

The present results clearly show that the metal-based intelligent composite with embedded SMA wires can be successfully fabricated by our proposed synthesis approach. The insulation interface between the SMA and the metal matrix can be successfully developed, and the composite has a significant reduction effect on vibration. However, the endurance life of this type of composite requires extensive study and apparently will be a great challenge for both scientific research and industrial applications, since, as mentioned before, the SMA undergoes a volume expansion during the phase transformation. The relatively greater difference in the thermal expansion coefficient between the SMA and insulation oxide film will produce a very large internal stress in the oxide film, the wire, and the matrix, and may cause the oxide film to break after a certain period of service life. Therefore, (1) how to establish a strong bonding between the interface and the SMA wire, and between the interface and the matrix, and (2) how to identify good interface materials that can provide both strength and insulation become the most imposing problem in these types of metal matrix smart materials. This research

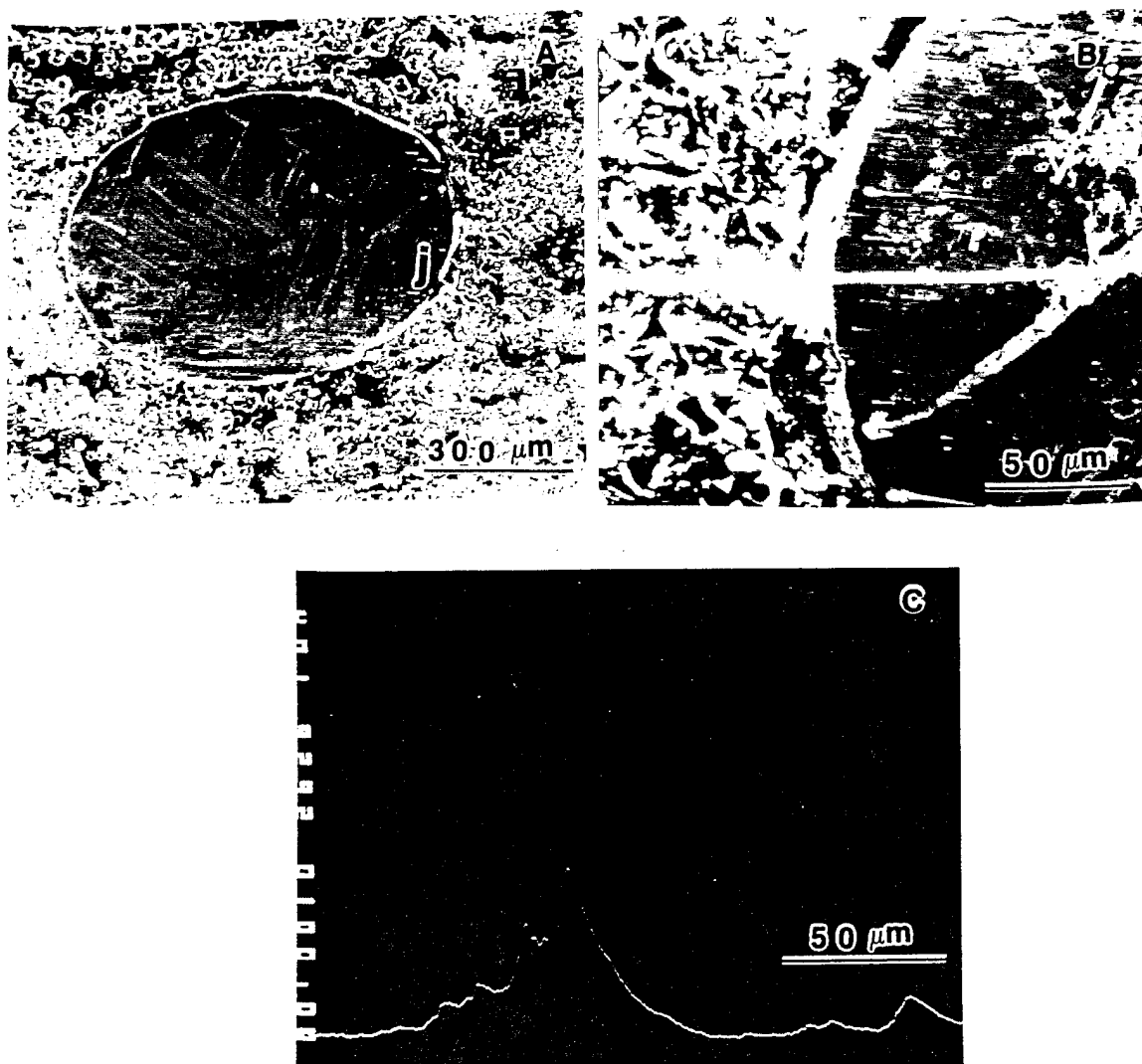


Figure 7. Microstructure of the NiTi wire embedded aluminum insulator film matrix composite wire,  $\text{Al}_2\text{O}_3$ .

- a. The low magnification optical microstructure.
- b. The microstructure near the insulator film.
- c. Oxygen distribution at the insulator film.

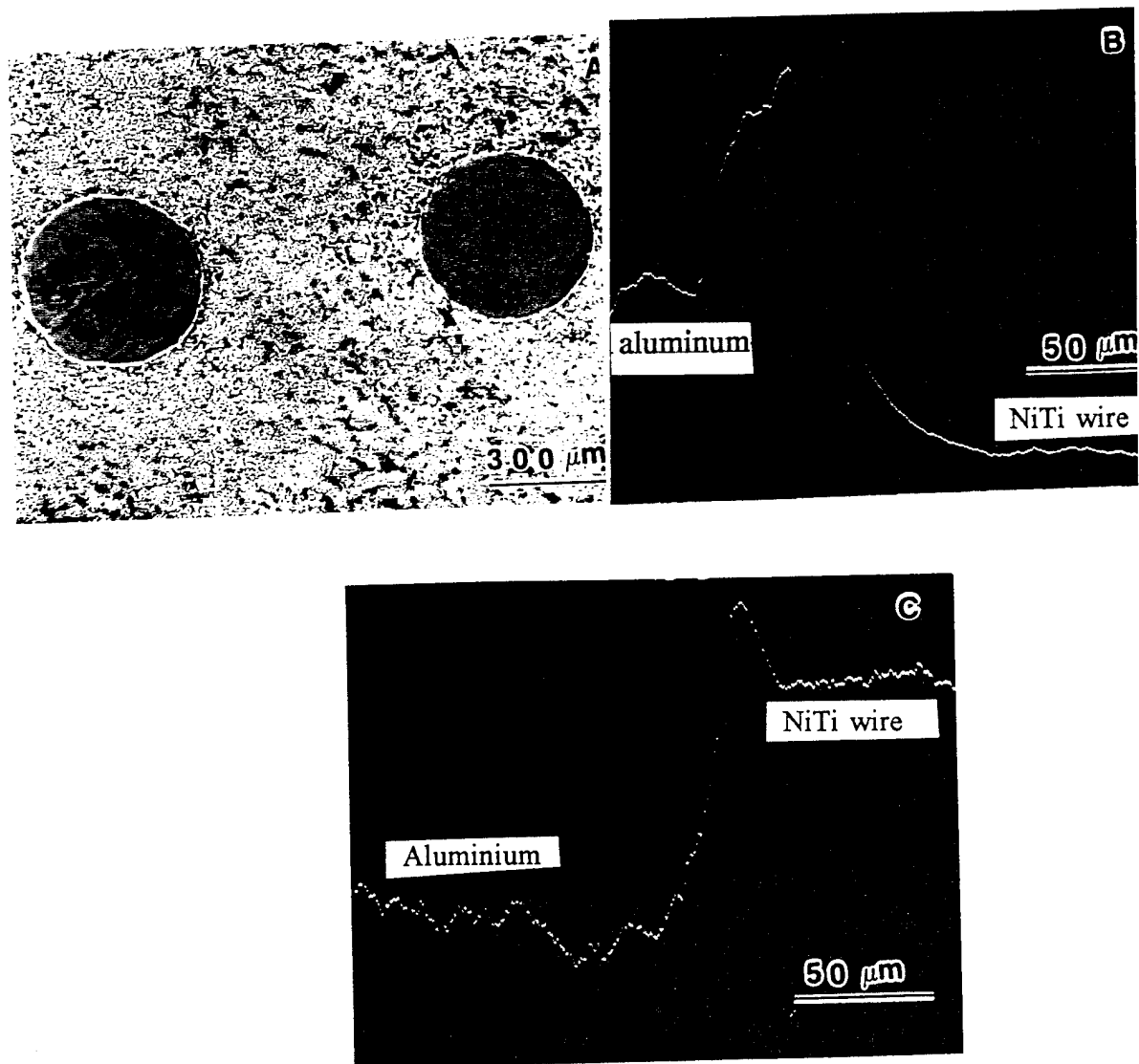


Figure 8. The microstructure of the Niti wire embedded aluminum matrix composite with Ta-containing oxide film.

- a. The low magnification microstructure.
- b. Oxygen distribution near the insulator film.
- c. Ta distribution near the insulator film.

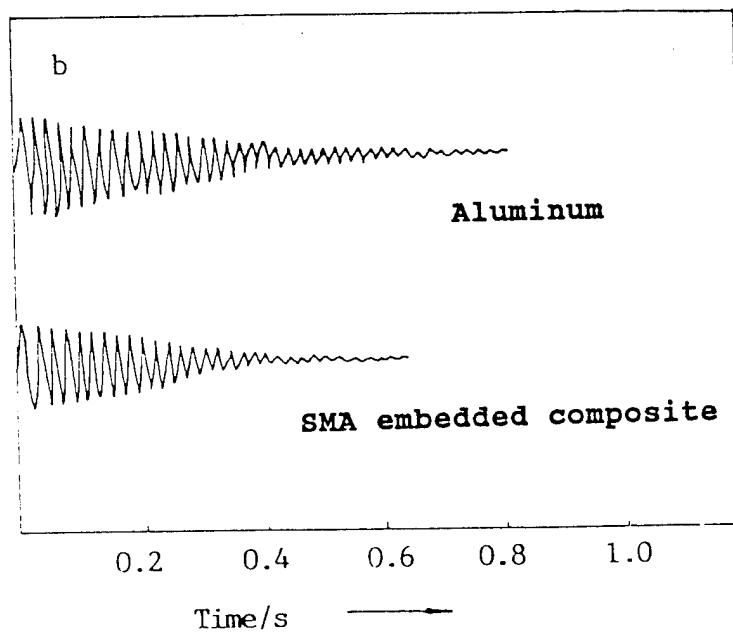


Figure 9. The vibration spectrum of high Aluminum alloy and the NiTi wire embedded composite.

is being continued in our laboratory.

One alternative way to solve this problem is by substituting the martensite to austenite transformation in the SMA by the R-phase austenite transformation, because an R-phase to austenite transformation produces much less volume change than that of the martensite to austenite transformation. For example, the martensite to austenite transformation in the NiTi alloy produced a 0.5% volume change, while the R-phase to austenite transformation barely produces a volume change [1,7]. Since the R-phase to austenite transformation also produces a great difference in Young's modulus, this type of composite is assumed to provide the same level of vibration reduction effect as the SMA composites currently studied.

The key issue for the substitution of the ordinary SMA wire by an R-phase to austenite transformation SMA is to design or choose a suitable SMA material in which the transformation temperature of the R-phase to austenite is much larger than that of martensite to R-phase transformation. This may be achieved through a careful selection of the composition of the SMA materials and a well-controlled heat treatment process.

## SUMMARY

Aluminum-based intelligent composites with embedded SMA wires have been successfully fabricated using various thin film deposition techniques. By using the present proposed process, the SMA and aluminum matrix can be well insulated through an insulation oxide film, which demonstrates a strong adhesion with the SMA. The strong bonding between the interface layer and the SMA wire is established through the composition gradient layer, which provides a transition zone between the insulation film and the SMA. The vibration test proves that this metal-based composite can effectively reduce vibration, and hence, could be used as a potential smart material for various industries, as well as defense applications.

## REFERENCES

1. Furakubo, H. 1978. *Shape Memory Alloys* (Translated from Japanese), Gordon and Breach Science Publishers.
2. Maclean, B. J., G. J. Patterson and M. S. Misra, 1990. "Modeling of a Shape Memory Integrated Actuator for Vibration Control of Large Space Structures," *J. Intell. Mater. Sys. Struc.*, V2, p. 72.
3. Baz, A., K. Iman, and J. McCoy, 1990. "The Dynamics of Helical Shape Memory Actuators," *J. Intell. Mater. Sys. Struc.*, V1, p. 105.
4. Ikegami, R., D. G. Wilson, J. R. Anderson and G. J. Julien, 1990. "Active Vibration Control Using Nitinol and Piezoelectric Ceramic," *J. Intell. Mater. Sys. and Struc.*, V1, p. 184.

5. Breitbach, E. J., 1990. *Proceedings of the First Joint U.S./Japan Conference on Adaptive Structures*, Maui, Hawaii, p. 32.
6. Ohring, M., 1992. *The Material Science of Thin Film*, Academic Press, Inc., Harcourt Brace Jovanovich Publishers, p. 445.
7. Miyazaki, S., S. Kimura, and K. Otsuka, 1988. "Shape-Memory Effect and Pseudoelasticity Associated with the R-Phase Transition in Ti-50.5 at% Ni Single Crystals," *Phil Mag.* V57, p. 346.